

PATENT COOPERATION TREATY
PCT

INTERNATIONAL PRELIMINARY REPORT ON PATENTABILITY

(Chapter II of the Patent Cooperation Treaty)

(PCT Article 36 and Rule 70)

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Applicant's or agent's file reference FP19993	FOR FURTHER ACTION See Form PCT/IPEA/416	
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Applicant COMMONWEALTH SCIENTIFIC AND INDUSTRIAL RESEARCH ORGANISATION et al		

1. This report is the international preliminary examination report, established by this International Preliminary Examining Authority under Article 35 and transmitted to the applicant according to Article 36.

2. This REPORT consists of a total of 3 sheets, including this cover sheet.

3. This report is also accompanied by ANNEXES, comprising:

a. ☒ (sent to the applicant and to the International Bureau) a total of 17 sheets, as follows:

☒ sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications authorized by this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions).

☐ sheets which supersede earlier sheets, but which this Authority considers contain an amendment that goes beyond the disclosure in the international application as filed, as indicated in item 4 of Box No. I and the Supplemental Box.

b. ☐ (sent to the International Bureau only) a total of (indicate type and number of electronic carrier(s)) , containing a sequence listing and/or table related thereto, in computer readable form only, as indicated in the Supplemental Box Relating to Sequence Listing (see Section 802 of the Administrative Instructions).

4. This report contains indications relating to the following items:

<input checked="" type="checkbox"/> Box No. I	Basis of the report
<input type="checkbox"/> Box No. II	Priority
<input type="checkbox"/> Box No. III	Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
<input type="checkbox"/> Box No. IV	Lack of unity of invention
<input checked="" type="checkbox"/> Box No. V	Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement
<input type="checkbox"/> Box No. VI	Certain documents cited
<input type="checkbox"/> Box No. VII	Certain defects in the international application
<input type="checkbox"/> Box No. VIII	Certain observations on the international application

Date of submission of the demand 4 May 2005	Date of completion of the report 18 October 2005
Name and mailing address of the IPEA/AU AUSTRALIAN PATENT OFFICE PO BOX 200, WODEN ACT 2606, AUSTRALIA E-mail address: pct@ipaaustralia.gov.au Facsimile No. (02) 6285 3929	Authorized Officer ROGER HOWE Telephone No. (02) 6283 2159

INTERNATIONAL PRELIMINARY REPORT ON PATENTABILITY

International application No.

PCT/AU2004/000899

Box No. I Basis of the report

1. With regard to the language, this report is based on the international application in the language in which it was filed, unless otherwise indicated under this item.
 - ☐ This report is based on translations from the original language into the following language which is the language of a translation furnished for the purposes of:
 - ☐ international search (under Rules 12.3 and 23.1 (b))
 - ☐ publication of the international application (under Rule 12.4)
 - ☐ international preliminary examination (under Rules 55.2 and/or 55.3)
2. With regard to the elements of the international application, this report is based on (*replacement sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to this report*):
 - ☐ the international application as originally filed/furnished
 - ☒ the description:
 - pages 1-3, 12-25 as originally filed/furnished
 - pages* 4-11a received by this Authority on 14 October 2005 with the letter of 14 October 2005
 - pages* received by this Authority on with the letter of
 - ☒ the claims:
 - pages as originally filed/furnished
 - pages* as amended (together with any statement) under Article 19
 - pages* 26-33 received by this Authority on 14 October 2005 with the letter of 14 October 2005
 - pages* received by this Authority on with the letter of
 - ☒ the drawings:
 - pages 1/8-8/8 as originally filed/furnished
 - pages* received by this Authority on with the letter of
 - pages* received by this Authority on with the letter of
 - ☐ a sequence listing and/or any related table(s) - see Supplemental Box Relating to Sequence Listing.
3. ☐ The amendments have resulted in the cancellation of:
 - ☐ the description, pages
 - ☐ the claims, Nos.
 - ☐ the drawings, sheets/figs
 - ☐ the sequence listing (*specify*):
 - ☐ any table(s) related to the sequence listing (*specify*):
4. ☐ This report has been established as if (some of) the amendments annexed to this report and listed below had not been made, since they have been considered to go beyond the disclosure as filed, as indicated in the Supplemental Box (Rule 70.2(c)).
 - ☐ the description, pages
 - ☐ the claims, Nos.
 - ☐ the drawings, sheets/figs
 - ☐ the sequence listing (*specify*):
 - ☐ any table(s) related to the sequence listing (*specify*):

* If item 4 applies, some or all of those sheets may be marked "superseded."

INTERNATIONAL PRELIMINARY REPORT ON PATENTABILITY

International application No.

PCT/AU2004/000899

Box No. V Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Statement

Novelty (N)	Claims 1-45	YES
	Claims	NO
Inventive step (IS)	Claims 1-45	YES
	Claims	NO
Industrial applicability (IA)	Claims 1-45	YES
	Claims	NO

2. Citations and explanations (Rule 70.7)

Citations:

- D1: OKABE, et. al., "Reaction Pathways during Metallothermic Reduction of $TiCl_4$ by Utilizing Chemical Potential Diagrams," *Titanium Extracting and Processing*, editors B. Mishra and G. J. Kipouros, The Minerals, Metals and Materials Society, 1997, pp 133-150,
- D2: JP 52-078608
- D3: RU 2082561
- D4: DeKOCK, et. al., "Attempted Preparation of Ti-6-4 Alloy Powders from $TiCl_4$, Al, VCl_4 and Na," *Metallurgical Transactions B*, Vol. 18B, September 1987, pp 511-517
- D5: BARTLETT, et. al., "Titanium Powder by Magnesium Reduction of $TiCl$ in Liquid Zinc Alloy," *Light Metals 1994* (edited by U. Mannweiler), The Minerals, Metals and Materials Society, 1994, pp. 1181-1186.
- D6: US 5460642
- D7: US 5032176
- D8: WO 1992/014851 – see page 2 lines 1 to 12
- D9: EP 134643
- D10: DE 2747016 – see claim 1, page 4 lines 11 – 20 and 26 – 34.

Explanation:

None of the documents cited in the International Search Report disclose a method or apparatus for producing metal-aluminium compounds wherein titanium tetrachloride is reduced in a stepwise fashion to subchlorides and then reacted in a second step under higher temperature with aluminium to form $AlCl_3$ gas and titanium-aluminium compounds. While D1 discloses that the reaction pathway for reacting Al with $TiCl_4$ passes through subchloride intermediaries, and D2-D10 each disclose the reaction of metal halide (eg $TiCl_4$) with metal (eg Al) to produce intermetallics/alloys (eg Ti-Al) and metal halide (eg $AlCl_3$), there is no suggestion in these documents to conduct this reaction is a stepwise fashion, i.e. produce the subchlorides and then react them to form the final products. D11 does disclose a stepwise reaction, with $TiCl_4$ being reacted with Ti to form subchlorides, which are then dissolved in molten salt and reacted with magnesium to form titanium powder. While it would be obvious to a person skilled in the art to substitute the aluminium in the initial reaction with other reductants, and indeed claims 33 and 34 as amended, for example, define such a substitution, it would not be obvious to replace the aluminium of the second step with magnesium since the aim of the invention is to produce metal-aluminium alloys or intermetallics. Thus the teachings of D11 can not be combined with D1-D10 to arrive at the claimed methods. Likewise, there is no suggestion of using a stepwise process to produce vanadium or zirconium metals, alloys or intermetallics by treating the respective subhalides with aluminium, and it does not appear obvious to follow the teachings of D1-D11 to do so.

Therefore all claims are Novel and Inventive in light of the above documents.

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development and is yet to be demonstrated on an industrial scale.

Attempts have been made to use aluminium as a reducing agent for TiCl_4 in plasma systems. For reduction of TiCl_4 using aluminium, the products are in the form of solid phase titanium-aluminium intermetallic compounds mixed with aluminium chloride and some residual titanium dichloride. A description of various attempts using aluminium together with a description of the thermodynamics of the process are given by Murphy and Bing (*High Temp. Chem. Processes*, Vol 3, 365-374, 1994). Because of difficulties associated with gas phase reactions it has not been possible to produce titanium and/or titanium-aluminium compounds by direct aluminothermic reduction of titanium chlorides.

Summary of the Invention

In accordance with a first aspect, the present invention provides a stepwise method of producing titanium-aluminium compounds, comprising a first step of:

- reducing an amount of titanium chloride (TiCl_4) with an amount of aluminium at a temperature to trigger reactions to form titanium subchloride(s) and aluminium chloride (AlCl_3) products;

and then a second step of:

- mixing said products, with the addition of more aluminium if required, and heating the mixture in a reaction zone to a temperature above 300°C to form AlCl_3 in a gas phase, and to produce an end product in the reaction zone of the titanium-aluminium compounds.

When the term titanium subchloride is used, it can refer to titanium trichloride TiCl_3 and/or titanium dichloride TiCl_2 , or other combinations of titanium and chloride excluding TiCl_4 which is referred to herein as titanium chloride.

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When the term titanium compound is used, it can refer to titanium alloys and/or titanium/metal intermetallic compounds. In one preferred form which is referred to herein, the titanium compounds include titanium-aluminium
5 alloys and/or titanium-aluminium intermetallic compounds.

In one embodiment, the method can also comprises the step of driving the removal of AlCl_3 away from the reaction zone to favour a forward reaction in the second step. In one form, the step of removing the AlCl_3 from the reaction
10 zone is continuous.

In one embodiment, the first step can be conducted at a temperature above the boiling point of AlCl_3 . In another embodiment, the first step can be conducted at a temperature above 200C.

15 In one embodiment of the method, the first step can be conducted with an excess amount of aluminium present to reduce all of the titanium chloride (TiCl_4) to form said titanium subchloride(s) and aluminium chloride (AlCl_3) products. In one embodiment, TiCl_3 is prepared by the
20 reduction of TiCl_4 , although this reaction may also form titanium dichloride TiCl_2 .

In one embodiment of the method, the second step can be conducted at a temperature in the range 300C to 1000C.

One embodiment of the method can comprise the further
25 step of recycling at least some of the aluminium chloride formed, and utilising the aluminium chloride to produce TiCl_4 . The aluminium chloride can be used to reduce titanium oxide to produce TiCl_4 .

In any of the embodiments of the method mentioned,
30 aluminium oxide can be produced by the reduction of titanium oxide and the aluminium oxide electrolysed to produce aluminium raw material for use in the steps of said method.

In one embodiment of the method, the aluminium
35 chlorides can be condensed away from the reaction zone at a temperature lower than that in the reaction zone. In one embodiment of the method, if titanium subchloride

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escapes the reaction zone it can be condensed at a temperature different to that in the reaction zone. Furthermore the condensed titanium subchloride can optionally be returned to the reaction zone.

5 In further embodiments of the method, the precursor material can include a source of one or more elements selected from the group comprising chromium (Cr), niobium (Nb), vanadium (V), zirconium (Zr), silicon (Si), boron (B), molybdenum (Mo), tantalum (Ta) and carbon (C), and
10 products of said method can include titanium-aluminium compounds which include one or more of these elements. The source of the element(s) can be a metal halide, a subhalide, a pure element or another compound which includes the element. The products can also include one
15 or more of an intermetallic compound, a titanium-(selected element)-alloy, and intermediate compounds. The source may also include a source of other precursors containing a required alloy additive, depending upon the required end product.

20 In one form of the method, the source can include vanadium subchloride, such as vanadium trichloride and/or vanadium dichloride, and a product of said method is an alloy or intermetallic complex including titanium, aluminium and vanadium. This method can comprise the
25 steps of adding the source in appropriate proportions, and carrying out the method to produce Ti-6Al-4V.

 In a further form of the method, the source can include zirconium subchloride, and a product of the method is an alloy or intermetallic complex including titanium,
30 aluminium, zirconium and vanadium.

 In one form of the method, the source can include niobium halide and chromium halide, and a product of said method is an alloy or intermetallic complex including titanium, aluminium, niobium and chromium. This method
35 can also comprise the step of adding the source in appropriate proportions, and carrying out the method to produce Ti-48Al-2Nb-2Cr.

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In one embodiment of the method, the aluminium can be added in the form of a powder having an approximate upper grain size of less than about 50 micrometres. In an alternative form of the method, the aluminium can be in the form of a powder of an approximate upper grain size of greater than about 50 micrometres; and the method comprises the step of milling the aluminium powder and titanium subchloride to reduce the grain size of the aluminium powder in at least one dimension. In yet another alternative form of the method, the aluminium can be in the form of flakes having a thickness in one dimension of less than about 50 micrometres. Using a fine aluminium powder is preferred, although the relatively coarser aluminium powder or flakes is a cheaper raw material.

In an embodiment, the method is conducted in an inert gas atmosphere or in a vacuum.

In any of the embodiments described, the method can also include a pre-processing step of forming the titanium subchloride as precursor material. The inventor has found that using a precursor material which includes titanium subchloride (preferably titanium trichloride) gives a number of advantages. There are not the problems of different, uncontrollable phases which are involved in starting from titanium tetrachloride as a precursor. The composition of the end product is relatively controllable and depends on the ratios of the starting materials. The correct ratios of starting materials are incorporated in the precursor materials to produce the appropriate proportions of components in the product.

The inventor believes that the new method enables a cheaper and more controllable process for the production of titanium-aluminium compounds. It is not necessary to convert the raw titanium minerals to titanium metal, as in some of the prior art processes discussed earlier. In one embodiment of the present process, titanium oxide can be chlorinated using conventional technology to give titanium

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tetrachloride. This can then be reduced using aluminium or hydrogen to give titanium subchlorides (mainly titanium trichloride), which can then be used as the precursor material for the formation of the titanium-aluminium compounds.

It is possible to form Ti-6Al-4V using this process, which is one of the major titanium alloys used. It is also possible to form Ti-48Al-2Nb-2Cr. It is also possible to form other alloys such as Ti-Al-Nb-C, and Ti₃Al based alloys.

A process in accordance with embodiments of the present invention described has the advantage that alloy powder is produced directly, with no further physical processing.

In accordance with a second aspect, the present invention provides a method for production of a powder of titanium-aluminium intermetallic compounds including at least one of Ti₃Al, TiAl and TiAl₃, and alloys based on titanium-aluminium intermetallics, as defined in the first aspect, and wherein starting materials for the method include aluminium powder and titanium chloride.

In accordance with a third aspect, the present invention provides a method of producing titanium-aluminium compounds, comprising a first step of:

- heating an amount of titanium chloride (TiCl₄) in a plasma of an inert gas and hydrogen mixture, to produce titanium subchloride(s);

and then a second step of:

- mixing aluminium with said titanium subchloride(s), and heating the resultant mixture to produce titanium-aluminium compounds and AlCl₃.

In one embodiment, the method of the third aspect can be otherwise as defined in the first aspect.

In accordance with a fourth aspect, the present invention provides a stepwise method of producing titanium-aluminium compounds, comprising a first step of:

- reducing an amount of titanium chloride (TiCl₄)

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with hydrogen in an inert gas atmosphere or in a vacuum, and at a temperature to trigger reactions to form titanium subchloride(s) and aluminium chloride (AlCl_3) products;

5 and then a second step of:

- mixing said products with aluminium, and heating the mixture in a reaction zone to a temperature above 300C to form AlCl_3 in a gas phase, and to produce an end product in the reaction zone of the titanium-aluminium compounds.

10 In one embodiment, the method of the fourth aspect can be otherwise as defined in the first aspect.

In accordance with a fifth aspect, the present invention provides a stepwise method of producing

- 15 titanium-aluminium compounds, comprising a first step of:
- heating a mixture of TiCl_4 and aluminium to form products TiCl_3 and AlCl_3 , at a temperature less than 300C;

and then a second step of:

- 20
- mixing said products, with the addition of more aluminium if required, and heating the mixture to a reaction zone temperature above 300C to cause AlCl_3 to be evaporated from the reaction zone and to form titanium-aluminium compounds.

25 In one embodiment, the method of the fifth aspect can be otherwise as defined in the first aspect.

In accordance with a sixth aspect, the present invention provides a stepwise method of producing a metal-aluminium compound, comprising the first step of:

- 30
- adding a reducing agent to reduce an amount of a metal halide to form metal subhalide(s);

and the second step of:

- 35
- mixing said metal subhalide(s) with aluminium, and heating the mixture in a reaction zone to a temperature above 300C to form aluminium halides in a gas phase, and to produce an end product in the reaction zone comprising a metal compound

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containing a percentage of aluminium.

In one embodiment of this method, the reducing agent can be selected from the group comprising zinc, magnesium, sodium, aluminium or other like metals. In one embodiment
5 the metal halide can be a titanium subhalide such as titanium trichloride, and a product of the reaction can include titanium compounds.

In one embodiment, the method of the sixth aspect can be otherwise as defined in the first aspect.

10 In any of the embodiments described, the method can also comprise the further step of adding a reagent to a product of the method, to produce a further product.

In accordance with a seventh aspect, the present invention provides a method for the production of vanadium
15 and/or vanadium compounds, comprising the steps of mixing aluminium with a precursor material including vanadium subhalide, and heating the mixture, to form aluminium halides and vanadium and/or vanadium compounds.

20 In one embodiment, the vanadium compounds may include vanadium-aluminium alloys and/or vanadium aluminium intermetallic complexes.

In accordance with an eighth aspect, the present invention provides a method for the production of zirconium and/or zirconium compounds, comprising the steps
25 of mixing aluminium with a precursor material including zirconium subhalide, and heating the mixture, to form aluminium halides and zirconium and/or zirconium compounds.

30 In one embodiment, the zirconium compounds may include zirconium-aluminium alloys and/or zirconium-aluminium intermetallic complexes.

In accordance with a ninth aspect, the present invention provides an apparatus for the production of a metal compound, comprising:

- 35
- a reaction vessel arranged in use for the mixing of aluminium with a metal halide or subhalide;
 - the vessel also adapted in use for the resultant

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mixture to be heated to a temperature sufficient for the metal halide or subhalide to react with the aluminium to form the metal compound and an aluminium halide;

- 5 - one condensation zone arranged in use to operate at a temperature such that any metal halide or subhalide escaping the reaction mixture condenses in that condensation zone; and
- 10 - another condensation zone arranged in use to operate at a temperature such that the aluminium halide condenses in the another condensation zone.

In one embodiment, the apparatus also can comprise a third condensation zone arranged to condense metal halide that is produced by disproportionation from escaping the reaction mixture. In a further embodiment, the one
15 condensation zone can be arranged to return condensed metal halide or subhalide to the reaction zone.

In one embodiment, the reaction zone operates at a temperature T1 and the first condensation zone at a
20 temperature T2 which is lower than the temperature T1. In one form, the second condensation zone operates at a temperature T3 which is between T1 and T2.

In one example, the precursor material may be a material containing titanium as a component, such as
25 titanium trichloride and/or titanium dichloride. Where the precursor material includes titanium trichloride and where the apparatus includes a first condensation zone that operates at temperature T2, T2 is preferably below 200°C, wherein gaseous aluminium trichloride emanating from
30 the reaction zone is condensed.

When the apparatus includes a second condensation zone, T3 is below 500°C and titanium trichloride which escapes from the reaction zone is recondensed at the second condensation zone. In one embodiment, the second
35 condensation zone is located between the reaction zone and the first condensation zone.

Typically the apparatus includes a heating

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arrangement for heating the precursor material. In some
embodiments, openings are provided for the introduction of
further gases. Openings may also be provided to evacuate
the vessel to a low pressure. In still further
5 embodiments, the reaction vessel may be a number of
discrete vessels, each vessel providing a different
reaction or condensation zone.

Preferably, the apparatus of this aspect of the
invention is suitable for implementing the method of any
10 of the foregoing or following aspects of the invention
described herein.

In accordance with a tenth aspect, the present
invention provides an apparatus for the production of at
least one of a titanium compound, another metal compound
15 or a product, when the apparatus is used with the method
as defined in any one of the first to the eighth aspects.

In accordance with an eleventh aspect, the present
invention provides at least one of a titanium compound, a
metal compound or a product produced by either the
20 apparatus or the method as defined in any one of the first
to the ninth aspects.

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CLAIMS

1. A stepwise method of producing titanium-aluminium compounds, comprising a first step of:
 - 5 - reducing an amount of titanium chloride (TiCl_4) with an amount of aluminium at a temperature to trigger reactions to form titanium subchloride(s) and aluminium chloride (AlCl_3) products; and then a second step of:
 - 10 - mixing said products, with the addition of more aluminium if required, and heating the mixture in a reaction zone to a temperature above 300°C to form AlCl_3 in a gas phase, and to produce an end product in the reaction zone of the titanium-aluminium compounds.
2. A method as claimed in claim 1, wherein the method also provides for driving the removal of AlCl_3 from the reaction zone to favour a forward reaction in the second step.
3. A method as claimed in claim 2, wherein the removal of AlCl_3 from the reaction zone is continuous.
- 25 4. A method as claimed in any one of the preceding claims, wherein the first step is conducted at a temperature above the boiling point of AlCl_3 .
5. A method as claimed in any one of the preceding claims, wherein the first step is conducted at a temperature above 200°C .
- 30 6. A method as claimed in any one of the preceding claims, wherein the first step is conducted with an excess amount of aluminium present to reduce all of the titanium chloride (TiCl_4) to form said titanium subchloride(s) and aluminium chloride (AlCl_3)
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products.

- 5 7. A method as claimed in any one of the preceding claims, wherein the second step is conducted at a temperature in the range 300C to 1000C.
- 10 8. A method as claimed in any one of the preceding claims, comprising the further step of recycling at least some of the aluminium chloride formed, and utilising the aluminium chloride to produce $TiCl_4$.
- 15 9. A method as claimed in claim 8, wherein the aluminium chloride is used to reduce titanium oxide to produce $TiCl_4$.
- 20 10. A method as claimed in claim 9, wherein aluminium oxide is produced by reduction of titanium oxide, and the aluminium oxide is electrolysed to produce aluminium raw material for use in the method of any one of the preceding claims.
- 25 11. A method as claimed in any one of the preceding claims, wherein the aluminium chloride is condensed away from the reaction zone at a temperature lower than that in the reaction zone.
- 30 12. A method as claimed in any one of the preceding claims, wherein titanium subchloride which escapes the reaction zone is condensed at a temperature different to that in the reaction zone.
- 35 13. A method as claimed in claim 12, comprising the further step of returning the condensed titanium subchloride to the reaction zone.
14. A method as claimed in any one of the preceding claims, also comprising the step of introducing a

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- 5 source of one or more elements selected from the group comprising chromium, niobium, vanadium, zirconium, silicon, boron, molybdenum, tantalum and carbon, and products of said method include titanium-aluminium compounds which include one or more of these elements.
- 10 15. A method as claimed in claim 14, wherein the source of the element(s) can be a metal halide, a subhalide, a pure element or another compound which includes the element.
- 15 16. A method as claimed in claim 14 or claim 15, wherein the products also include one or more of an intermetallic compound, a titanium-(selected element)-alloy, and intermediate compounds.
- 20 17. A method as claimed in any one of claim 14 to claim 16, wherein the source includes vanadium subchloride, and a product of said method is an alloy or intermetallic complex including titanium, aluminium and vanadium.
- 25 18. A method as claimed in claim 17, comprising the steps of adding the source in appropriate proportions, and carrying out the method to produce Ti-6Al-4V.
- 30 19. A method as claimed in claim 14, wherein the source includes zirconium subchloride, and a product of the method is an alloy or intermetallic complex including titanium, aluminium, zirconium and vanadium.
- 35 20. A method as claimed in any one of claims 14 to 16, wherein the source includes niobium halide and chromium halide, and a product of said method is an alloy or intermetallic complex including titanium, aluminium, niobium and chromium.

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21. A method as claimed in claim 20, comprising the step of adding the source in appropriate proportions, and carrying out the method to produce Ti-48Al-2Nb-2Cr.
- 10
22. A method as claimed in any one of the preceding claims, wherein the aluminium is added in the form of a powder having an approximate upper grain size of less than about 50 micrometres.
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23. A method as claimed in any one of claims 1 to 21, wherein the aluminium is in the form of a powder of an approximate upper grain size of greater than about 50 micrometres, and the method comprises the step of milling the aluminium powder and titanium subchloride to reduce the grain size of the aluminium powder in at least one dimension.
- 20
24. A method as claimed in any one of claims 1 to 21, wherein the aluminium is in the form of flakes having a thickness in one dimension of less than about 50 micrometres.
- 25
25. A method as claimed in any one of the preceding claims, wherein the method is conducted in an inert gas atmosphere or in a vacuum.
- 30
26. A method for production of a powder of titanium-aluminium intermetallic compounds including at least one of Ti_3Al , $TiAl$ and $TiAl_3$, and alloys based on titanium-aluminium intermetallics as claimed in any one of claims 1 to 25, wherein starting materials for the method include aluminium powder and titanium chloride.
- 35
27. A method of producing titanium-aluminium compounds, comprising a first step of:

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- heating an amount of titanium chloride (TiCl_4) in a plasma of an inert gas and hydrogen mixture, to produce titanium subchloride(s);
and then a second step of:
 - mixing aluminium with said titanium subchloride(s), and heating the resultant mixture to produce titanium-aluminium compounds and AlCl_3 .
28. A method as claimed in claim 27 which is otherwise as claimed in any one of claims 2 to 25.
29. A stepwise method of producing titanium-aluminium compounds, comprising a first step of:
 - reducing an amount of titanium chloride (TiCl_4) with hydrogen in an inert gas atmosphere or in a vacuum, and at a temperature to trigger reactions to form titanium subchloride(s) and aluminium chloride (AlCl_3) products;
and then a second step of:
 - mixing said products with aluminium, and heating the mixture in a reaction zone to a temperature above 300C to form AlCl_3 in a gas phase, and to produce an end product in the reaction zone of the titanium-aluminium compounds.
30. A method as claimed in claim 29 which is otherwise as claimed in any one of claims 2 to 25.
31. A stepwise method of producing titanium-aluminium compounds, comprising a first step of:
 - heating a mixture of TiCl_4 and aluminium to form products TiCl_3 and AlCl_3 , at a temperature less than 300C;
and then a second step of:
 - mixing said products, with the addition of more aluminium if required, and heating the mixture to a reaction zone temperature above 300C to cause

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AlCl_3 to be evaporated from the reaction zone and to form titanium-aluminium compounds.

- 5 32. A method as claimed in claim 31 which is otherwise as claimed in any one of claims 2 to 25.
- 10 33. A stepwise method of producing a metal-aluminium compound, comprising the first step of:
- adding a reducing agent to reduce an amount of a metal halide to form metal subhalide(s);
- and the second step of:
- mixing said metal subhalide(s) with aluminium, and heating the mixture in a reaction zone to a temperature above 300C to form aluminium halides in a gas phase, and to produce an end product in the reaction zone comprising a metal compound containing a percentage of aluminium.
- 15
- 20 34. A method in accordance with claim 33, wherein the reducing agent is selected from the group comprising zinc, magnesium, sodium, aluminium or other like metals.
- 25 35. A method as claimed in claim 33 or claim 34 which is otherwise as claimed in any one of claims 2 to 25.
- 30 36. A method in accordance with any one of the preceding claims, comprising the further step of adding a reagent to a product of the method to produce a further product.
- 35 37. A method for the production of vanadium and/or vanadium compounds, comprising the steps of mixing aluminium with a precursor material including vanadium subhalide, and heating the mixture, to form aluminium halides and vanadium and/or vanadium compounds.

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- 5 38. A method in accordance with claim 37, wherein the vanadium compounds may include vanadium-aluminium alloys and/or vanadium aluminium intermetallic complexes.
- 10 39. A method for the production of zirconium and/or zirconium compounds, comprising the steps of mixing aluminium with a precursor material including zirconium subhalide, and heating the mixture, to form aluminium halides and zirconium and/or zirconium compounds.
- 15 40. A method in accordance with claim 38, wherein the zirconium compounds may include zirconium-aluminium alloys and/or zirconium-aluminium intermetallic complexes.
- 20 41. An apparatus for the production of a metal compound, comprising:
- a reaction vessel arranged in use for the mixing of aluminium with a metal halide or subhalide;
 - the vessel also adapted in use for the resultant mixture to be heated to a temperature sufficient for the metal halide or subhalide to react with the aluminium to form the metal compound and an aluminium halide;
 - one condensation zone arranged in use to operate at a temperature such that any metal halide or subhalide escaping the reaction mixture condenses in that condensation zone; and
 - another condensation zone arranged in use to operate at a temperature such that the aluminium halide condenses in the another condensation zone.
- 35 42. An apparatus as claimed in claim 41, also comprising a third condensation zone arranged to condense metal

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halide that is produced by disproportionation from escaping the reaction mixture.

- 5 43. An apparatus as claimed in claim 41 or claim 42, wherein the one condensation zone is arranged to return condensed metal halide or subhalide to the reaction zone.
- 10 44. An apparatus for the production of at least one of a titanium compound, another metal compound or a product, when the apparatus is used with the method as claimed in any one of claims 1 to 40.
- 15 45. A titanium compound, a metal compound or a product produced by either the apparatus or the method as claimed in any one of the preceding claims.